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METHODS OF CHEMICAL ANALYSIS OF RUTILE

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
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METHODS OF CHEMICAL ANALYSIS OF RUTILE

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METHODS OF CHEMICAL ANALYSIS OF RUTILE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 30 April 1967, after the draft finalized by the Methods of Chemical Analysis Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 Rutile is recovered while processing the beach sands. Besides its use in the paint industry and manufacture of arc welding electrodes, it is also used for the manufacture of ferro-titanium. In order to determine correctly the chemical composition of rutile, need was felt to prescribe standard methods of chemical analysis, so that these may primarily serve as referee methods and may also be used by laboratories for routine analysis. Due consideration has been given in the preparation of this standard to the facilities available in the country for such analysis.

0.3 In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard covers methods of chemical analysis of rutile in the composition ranges given below:

Elements	*Percent*
Titania (TiO_2)	90 and above
Silica (SiO_2)	Up to 2
Ferric oxide (Fe_2O_3)	Up to 2
Zirconia (ZrO_2)	Up to 3
Alumina (Al_2O_3)	Up to 1
Phosphorus Pentoxide (P_2O_5)	Up to 0·1

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*Rules for rounding off numerical values (*revised*).

2. SAMPLING

2.1 Samples shall be drawn and prepared in accordance with IS : 4166-1967*.

3. QUALITY OF REAGENTS

3.1 'Pure chemicals' and distilled water (*see* IS : 1070-1961†) shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis. Generally 'Guaranteed Reagents' grade or 'Analytical Reagent' grade chemicals are satisfactory.

4. GENERAL

4.1 In the methods prescribed in this standard, only relative number of Whatman filter papers have been prescribed. However, any other suitable brand of filter paper of corresponding porosity may also be used.

5. DETERMINATION OF SILICA BY THE GRAVIMETRIC METHOD

5.1 Outline of the Method — The sample is fused with potassium pyrosulphate. The melt is dissolved and evaporated with sulphuric acid to fumes, diluted and filtered. Residue is fused with sodium carbonate and the melt dissolved and evaporated with sulphuric acid as earlier, and filtered. The silica is ignited and determined by the loss in weight after volatilization with hydrofluoric acid in the presence of sulphuric acid.

5.2 Reagents

5.2.1 *Potassium Pyrosulphate* — solid.

5.2.2 *Dilute Sulphuric Acid* — 1 : 1 (*v/v*).

5.2.3 *Sodium Carbonate* — solid.

5.2.4 *Hydrofluoric Acid* — 40 percent.

5.3 Procedure

5.3.1 Transfer 0·5 g of an accurately weighed sample in a large platinum crucible containing about 10 to 15 g of potassium pyrosulphate, heat at a low temperature till potassium pyrosulphate is melted. Increase the temperature gradually to 700°C. As soon as the sample is completely decomposed, remove the crucible from heat, cover with lid, and allow it to cool to room temperature.

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*Methods of sampling ilmenite and rutile (*under preparation*).

†Specification for water, distilled quality (*revised*).

5.3.2 Transfer the crucible with its contents into a porcelain basin and extract the contents with 100 ml of dilute sulphuric acid. Transfer the basin to a hot water bath. After the melt is completely dissolved, remove the crucible and lid, washing down with dilute sulphuric acid and evaporate the solution to dense white fumes. Continue fuming for 30 minutes.

5.3.3 Cool the basin, dilute the solution to 50 ml with dilute sulphuric acid and filter through No. 40 filter paper. Remove any adhering particles from the basin by means of dilute sulphuric acid and finally with hot water until acid free, collecting the filtrate and washing into a 500-ml volumetric flask.

5.3.4 Transfer the filter paper along with the residue to a platinum crucible, ignite and fuse it with five grams of sodium carbonate. Extract the residue with about 25 ml of dilute sulphuric acid in the basin and evaporate to dense white fumes. Repeat the fuming for 30 minutes. Dilute to 50 ml with dilute sulphuric acid and filter through No. 40 filter paper. Preserve the filtrate.

5.3.5 Ignite the filter along with the residue obtain under **5.3.4** in a platinum crucible at 1 050°C to constant weight. Moisten the residue with a few drops of dilute sulphuric acid and add 5 to 10 ml of hydrofluoric acid. Evaporate to dryness, ignite, cool and weigh. Fuse this residue with one gram of potassium pyrosulphate and extract with 20 to 30 ml of dilute sulphuric acid. Add to the filtrate obtained under **5.3.3** and **5.3.4** and make up the volume to mark. Preserve the solution.

5.3.6 Carry out a blank determination following the same procedure and using the same quantity of all the reagents but without the sample.

5.4 Calculation

$$\text{Silica, percent} = \frac{(A-B)}{C} \times 100$$

where

A = weight in g of silica obtained under **5.3.5**,

B = weight in g of silica obtained from the blank, and

C = weight in g of the sample taken.

6. DETERMINATION OF FERRIC OXIDE BY THE VOLUMETRIC METHOD

6.1 Outline of the Method — From an aliquot of the solution preserved under **5.3.5** ferrous iron is first oxidized and then reduced completely with stannous chloride, excess of which is destroyed by mercuric chloride. The reduced iron is titrated with standard potassium dichromate solution using sodium diphenylamine sulphonate solution as indicator.

6.2 Reagents

6.2.1 *Concentrated Nitric Acid* — sp gr 1·42 (conforming to IS : 264-1950*).

6.2.2 *Concentrated Ammonium Hydroxide* — 20 percent.

6.2.3 *Ammonium Chloride Solution* — 10 percent (*w*/*v*).

6.2.4 *Dilute Hydrochloric Acid* — 1 : 1 (*v*/*v*).

6.2.5 *Concentrated Hydrochloric Acid* — sp gr 1·16 (conforming to IS : 265-1962†).

6.2.6 *Stannous Chloride Solution* — Dissolve by heating 60 g of pure stannous chloride in a mixture of 400 ml of concentrated hydrochloric acid and 600 ml of water until the solution is complete. Cool, add a few pieces of granulated tin and preserve the solution in an air-tight bottle.

6.2.7 *Mercuric Chloride Solution* — Prepare a saturated solution of mercuric cholride in water.

6.2.8 *Sulphuric-Phosphoric Acid Mixture* — Add slowly 150 ml of concentrated sulphuric acid to 700 ml of water with continuous stirring. Add to this solution 150 ml of syrupy phosphoric acid. Destroy any oxidizable impurities by adding potassium permanganate solution (0·1 N) drop by drop until the pink colour of permanganate persists. Cool the solution.

6.2.9 *Sodium Diphenylamine Sulphonate Indicator Solution* — Dissolve 0·2 g of the reagent in 100 ml of hot water.

6.2.10 *Standard Potassium Dichromate Solution* — 0·1 N. Dry pure potassium dichromate at 105° to 110°C for one and a half hours. Cool in a desiccator and transfer exactly 4·904 g to a one-litre volumetric flask carefully through a funnel using water at room temperature. Wash the funnel thoroughly and dissolve the salt by shaking until the solution is complete. Make up to the mark and mix well.

6.3 Procedure

6.3.1 Pipette out exactly 100 ml of the solution preserved in **5.3.5** into 500-ml beaker. Add 5 ml of concentrated nitric acid and heat to expel brown fumes. Adjust the volume to about 100 ml. Add concentrated ammonium hydroxide until slightly but distinctly alkaline. Boil for few minutes and allow to settle for some time. Filter while hot through No. 41 filter paper and wash few times alternately with ammonium chloride solution and hot water. Dissolve the precipitate through the filter paper with 20 ml of dilute hydrochloric acid and wash thoroughly with hot water collecting the filtrate and washings in the original beaker. Evaporate to 40 ml and add 40 ml of concentrated hydrochloric acid. Reduce the iron by adding stannous chloride solution dropwise to the boiling solution until the solution becomes colourless. Add two to three drops of stannous chloride solution in excess. Cool the solution to room temperature.

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*Specification for nitric acid.

†Specification for hydrochloric acid (*revised*).

6.3.2 Add excess of mercuric chloride (about 5 ml) and shake the flask. At this stage a silky white precipitate appears. If it fails to appear or if a blackish precipitate appears, reject the test. If the precipitate is too much, add further 5 ml of mercuric chloride solution. Keep for about 2 to 5 minutes and add 15 ml of sulphuric-phosphoric acid mixture, and dilute to about 250 ml, washing down the sides of the flask. Add three to four drops of sodium diphenylamine sulphonate indicator solution and titrate slowly with standard potassium dichromate solution stirring continuously until the green colour begins to darken. Continue the titration slowly till a drop of the dichromate solution produces a stable violet-blue colouration.

6.3.3 Make a blank determination following the same procedure and using the same amounts of all reagents, but without the sample.

6.4 Calculation

$$\text{Ferric oxide, percent} = \frac{(A-B)\,C \times 7{\cdot}985}{D}$$

where

A = volume in ml of the standard potassium dichromate solution required for the test solution,

B = volume in ml of the standard potassium dichromate solution required for the blank,

C = normality of the standard potassium dichromate solution, and

D = weight in g of the sample represented by the aliquot taken.

7. DETERMINATION OF TITANIA BY THE REDUCTION (VOLUMETRIC) METHOD

7.1 Outline of the Method — After reduction with zinc amalgam, titanium is determined volumetrically by titrating with standard ferric ammonium sulphate solution using potassium thiocyanate as indicator.

7.2 Apparatus — The apparatus for reduction (Fig. 1) consists of a globular separating funnel *A* with a ground glass stopper *B*. Flask *C* of 50 ml capacity is attached to the lower end of the separating funnel by means of a pressure tubing *D* having a pinch cock *E*.

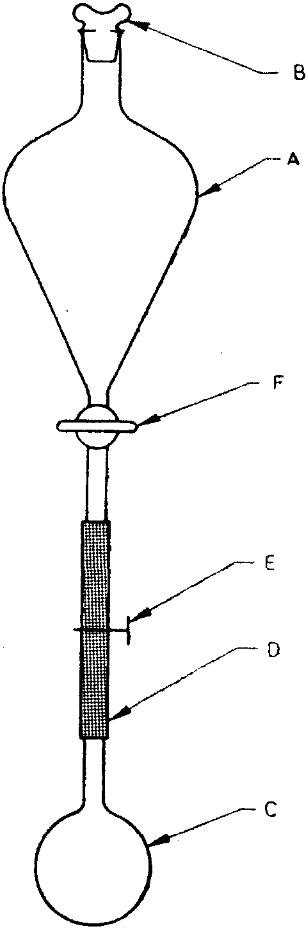

FIG. 1 APPARATUS FOR DETERMINATION OF TITANIA BY THE REDUCTION (VOLUMETRIC) METHOD

7.3 Reagents

7.3.1 *Dilute Sulphuric Acid* — one percent.

7.3.2 *Carbon Dioxide* — gas.

7.3.3 *Standard Ferric Ammonium Sulphate Solution* — 0·1 N. Dissolve 48 g of pure ferric ammonium sulphate in a mixture of 250 ml water containing 10 ml of concentrated sulphuric acid, add potassium permanganate solution dropwise till the pink colour disappears, and dilute to one litre. Standardize the solution after reduction with zinc amalgam following the procedure under **7.4** and titrating against standard potassium permanganate.

7.3.4 *Standard Potassium Permanganate* — 0·1 N. Dissolve 3·2 g of potassium permanganate in one litre of water. Keep the solution in a glass-stoppered amber-coloured bottle for five to six days. Filter through glass wool into a clean glass-stoppered bottle and standardize against sodium oxalate or oxalic acid (AR).

7.3.5 *Potassium Thiocyanate Solution* — saturated.

7.3.6 *Zinc Amalgam* — Wash 15 g pure fine mesh zinc with dilute sulphuric acid and heat with 300 g of pure mercury after adding 5 ml of sulphuric acid (1:4) for one hour on a water bath in a fume cupboard. (*Caution* : Mercury vapours are highly poisonous). Wash the amalgam several times with dilute sulphuric acid (1:1) after cooling, and separate the liquid portion from the solids in a separating funnel. Preserve the liquid amalgam under dilute sulphuric acid (1:4).

7.4 Procedure

7.4.1 Fill the flask *C*, the rubber tubing *D* and the stem up to the stop-cock *F* with freshly boiled and cooled water containing one percent of sulphuric acid. Close pinch-cock *E* and the stop-cock *F*. Pipette 100 ml of the solution preserved under **5.3.5** into the separating funnel *A* containing 50 ml of zinc amalgam. Pass carbon dioxide gas into the separating funnel for 5 minutes. Stopper the funnel and shake it for 5 minutes. Open stop-cock *F* and pinch-cock *E* and transfer amalgam into the flask *C* by pressing and releasing the pressure tubing *D*. Close the pinch-cock *E* and stop-cock *F* immediately after the last drop of amalgam has passed into the flask *C*. Disconnect the flask *C* from the pressure tubing *D*. Remove the stopper *B* and wash it with boiled and acidified water into the separating funnel. Add 5 ml of saturated potassium thiocyanate solution in the separating funnel and titrate rapidly with standard ferric ammonium sulphate solution till the appearance of wine red colour. Open stop-cock *F* and squeeze the rubber tubing to force the liquid into the separating funnel. Complete titration by adding standard ferric ammonium sulphate solution drop by drop till wine red colour is permanent.

7.4.2 Repeat the experiments with another 100-ml aliquot following the procedure under **7.4.1** and adding nearly whole of the volume of standard ferric ammonium sulphate solution required for the previous titration in the beginning of the titration; then agitate by shaking with a gentle rotary motion and continue till the final end point is reached.

7.4.3 Carry out a blank determination following the same procedure and using the same quantity of all reagents but without the sample.

7.5 Calculation

$$\text{Titania, percent} = \frac{(A-B)\,C \times 7{\cdot}99}{D}$$

where

A = volume in ml of the standard ferric ammonium sulphate solution used for the test solution,

B = volume in ml of the standard ferric ammonium sulphate solution used for the blank,

C = normality of the standard ferric ammonium sulphate solution, and

D = weight in g of the sample represented by the aliquot taken.

8. DETERMINATION OF ZIRCONIA BY THE GRAVIMETRIC METHOD

8.1 Outline of the Method — From a suitable aliquot of the solution, preserved under **5.3.5**, mixed oxides are precipitated. The precipitate is treated with sodium peroxide and ammonium hydroxide. After separating iron, zirconium is precipitated from the filtrate as phosphate and weighed.

8.2 Reagents

8.2.1 *Concentrated Nitric Acid — See* **6.2.1.**

8.2.2 *Concentrated Ammonium Hydroxide* — 20 percent.

8.2.3 *Ammonium Chloride* — solid.

8.2.4 *Methyl Red Indicator Solution* — 0·1 percent (*w/v*). Dissolve 0·1 g of methyl red in 3·72 ml of sodium hydroxide solution (0·1 N) and dilute to 250 ml with water. Filter if necessary.

8.2.5 *Ammonium Nitrate Solution* — 5 percent (*w/v*).

8.2.6 *Sodium Peroxide* — solid.

8.2.7 *Dilute Hydrochloric Acid* — 1:2 (*v/v*).

8.2.8 *Tartaric Acid* — solid.

8.2.9 *Hydrogen Sulphide* — gas.

8.2.10 *Ammonium Sulphide Solution* — 5:95 (*v/v*).

8.2.11 *Concentrated Sulphuric Acid* — sp gr 1·84 (conforming to IS : 266-1961*).

8.2.12 *Hydrogen Peroxide* — 20 volumes.

8.2.13 *Diammonium Phosphate* $(NH_4)_2$ HPO_4 — solid.

8.3 Procedure

8.3.1 Pipette out exactly 200 ml of the solution preserved under **5.3.5** into a 500-ml beaker, add 5 ml of concentrated nitric acid to oxidize ferrous iron and boil. Precipitate mixed oxides by adding concentrated ammonium hydroxide in presence of four to 5 g of ammonium chloride using methyl red indicator, until the colour changes to the distinct yellow. Allow to settle, filter and wash four times with hot ammonium nitrate solution. Digest the precipitate of mixed oxides with sodium peroxide for an hour on steam bath, dilute, filter and wash. Preserve the filtrate and washings for the determination of alumina.

8.3.2 Dissolve the precipitate in 25 ml of hot dilute hydrochloric acid and 5 g of tartaric acid. Dilute with water to a volume of 200 ml. Render the solution slightly ammoniacal, and pass in a rapid stream of hydrogen sulphide for 5 minutes. Digest at the side of the steam-bath (40°C) for about 15 to 30 minutes. Filter and wash with ammonium sulphide solution containing 5 g of ammonium chloride per litre. Discard the residue.

8.3.3 Adjust the acidity of the filtrate obtained under **8.3.2** so that the solution contains 10 percent by volume of sulphuric acid. Add sufficient hydrogen peroxide to complex all the titanium (an excess does no harm), and 0·5 g of diammonium phosphate, allow to stand at the side of the steam-bath (40°C) overnight, filter, wash with ammonium nitrate solution.

8.3.4 Transfer the paper and the residue to a platinum crucible and ignite very carefully so that the paper chars but does not flame. When the paper has charred, gradually increase the temperature until all carbon is gone and then heat at about 1050°C for 15 minutes. Cool in a desiccator and weigh as ZrP_2O_7.

8.4 Calculation

$$\text{Zirconia, percent} = \frac{A \times 46{\cdot}46}{B}$$

---

*Specification for sulphuric acid (*revised*).

where

A = weight in g of ZrP_2O_7 obtained under **8.3.4,** and

B = weight in g of the sample represented by the aliquot taken.

9. DETERMINATION OF ALUMINA BY THE OXINATE (VOLUMETRIC) METHOD

9.1 Outline of the Method — From the filtrate preserved under **8.3.1** aluminium is precipitated as oxinate from a buffered acetate solution and dissolved in hydrochloric acid followed by bromometric titration of the liberated 8-hydroxyquinoline.

9.2 Reagents

9.2.1 *Concentrated Hydrochloric Acid* — *See* **6.2.4**.

9.2.2 *Bromocresol Purple Indicator Solution* — 0·4 g/l. Dissolve 0·04 gram of bromocresol purple indicater in 100 ml of hot water, cool and filter.

9.2.3 *Dilute Ammonium Hydroxide* — 1:3 (*v*/*v*).

9.2.4 *8-Hydroxyquinoline 'Oxine' Solution* — Dissolve 5 g of 8-hydroxyquinoline in 15 ml of glacial acetic acid by warming and dilute with water to 250 ml.

9.2.5 *Ammonium Acetate* — solid.

9.2.6 *Potassium Bromide* — solid.

9.2.7 *Standard Potassium Bromate Solution* — 0·05 N. Dry some finely powdered (AR) potassium bromate for 1 to 2 hours at 120°C and allow to cool in a closed vessel in a desiccator. Weigh out accurately 1·392 g of the pure potassium bromate and dissolve it in one litre of water in a volumetric flask.

9.2.8 *Potassium Iodide Solution* — 10 percent (*w*/*v*).

9.2.9 *Standard Sodium Thiosulphate Solution* — 0·05 N. Weigh out 12·5 g of (AR) sodium thiosulphate crystals, dissolve in freshly boiled and cooled water and make up to one litre in a volumetric flask. If the solution is to be kept for more than a few days, add 0·1 g of sodium carbonate or three drops of chloroform.

9.2.10 *Starch Solution* — Make a suspension of one g of soluble starch in about 5 ml of water and add it carefully to 100 ml of boiling water. Cool the solution before use. Prepare fresh as needed.

9.3 Procedure

9.3.1 Acidify the filtrate preserved for determination of alumina under **8.3.1** with concentrated hydrochloric acid and concentrate the solution to about 150 ml. Cool, add three to four drops of bromocresol purple indicator solution and dilute ammonium hydroxide until the colour of the solution just turns violet. Keep the solution on a hot-plate. When the solution attains the temperature of 60° to 70°C, add 25 to 30 ml of oxine solution and 7 to 8 g of ammonium acetate slowly with stirring. Maintain the solution at 60° to 70°C until the precipitate is coagulated. Filter and wash the precipitate with hot water 10 to 12 times.

9.3.2 Dissolve the oxinate complex in warm concentrated hydrochloric acid and collect the solution in a 500-ml conical flask. Wash the filter with hot water till acid-free. Collect the washings also in the same conical flask.

9.3.3 Cool the solution add about 2 to 3 g of potassium bromide and shake till the solid dissolves. Now add exactly 20 ml of standard potassium bromate solution and keep for about 10 minutes for complete bromination. Add 10 ml of potassium iodide solution and titrate the liberated iodine with standard sodium thiosulphate solution using starch as indicator.

9.3.4 Pipette out 20 ml of standard bromate solution separately into a 500-ml conical flask. Add about 10 to 15 ml of potassium iodide solution followed by about 5 ml of concentrated hydrochloric acid. Dilute the contents to about 200 ml and titrate the liberated iodine against standard sodium thiosulphate solution using freshly prepared starch as indicator.

9.4 Calculation

$$\text{Alumina, percent} = \frac{(A-B) \times C \times 0{\cdot}425}{D}$$

where

A = volume in ml of the standard sodium thiosulphate solution required by the blank,

B = volume in ml of the standard sodium thiosulphate solution required by the unreacted bromate solution,

C = normality of the standard sodium thiosulphate solution, and

D = weight in g of the sample represented by the aliquot taken.

10. DETERMINATION OF PHOSPHORUS PENTOXIDE BY THE MOLYBDATE (ALKALIMETRIC) METHOD

10.1 Outline of the Method — The sample is fused with sodium peroxide in an iron crucible and melt extracted in water. From the filtrate, after separation of silica, phosphorus is co-precipitated with iron and then phosphorus precipitated as ammonium phosphomolybdate. The precipitate is filtered, washed and dissolved in excess of standard alkali. The unreacted alkali is back titrated against standard acid.

10.2 Reagents

10.2.1 *Sodium Peroxide* — solid.

10.2.2 *Sodium Hydroxide Solution* — 0·5 N. Dissolve 20 g of sodium hydroxide solution in water and make up the volume to one litre.

10.2.3 *Dilute Hydrochloric Acid* — 1:1 (*v/v*).

10.2.4 *Concentrated Hydrochloric Acid* — *See* **6.2.4**.

10.2.5 *Ferric Chloride Solution* — 10 percent (*w/v*).

10.2.6 *Concentrated Ammonium Hydroxide* — 20 percent.

10.2.7 *Concentrated Nitric Acid* — *See* **8.2.1**.

10.2.8 *Hydroxylamine Hydrochloride Solution* — 15 percent (*w/v*).

10.2.9 *Ammonium Nitrate* — solid.

10.2.10 *Ammonium Molybdate Solution* — Add solution *A* to solution *B* with constant stirring, then add 0·1 gram of ammonium phosphate dissolved in 10 ml of water and let stand for at least 24 hours before using. Keep the solution in a cool place and always filter before using. Prepare solutions *A* and *B* as follows:

a) *Solution A* — Dissolve 100 g of molybdic acid (85 percent MoO_3) in a mixture of 150 ml of concentrated ammonium hydroxide and 270 ml of water.

b) *Solution B* — Mix 537 ml of concentrated nitric acid and 1 280 ml of water and cool.

10.2.11 *Dilute Nitric Acid* — 1:99 (*v/v*).

10.2.12 *Potassium Nitrate Wash Solution* — one percent (*w/v*).

10.2.13 *Sodium Hydroxide Solution* — 0·1 N approximately (carbonate-free).

10.2.14 *Phenolphthalein Indicator Solution* — Dissolve 0·5 gram of the phenolphthalein in 100 ml of 60 percent of alcohol.

10.2.15 *Standard Hydrochloric Acid* — 0·1 N. Dilute about 11 ml of concentrated hydrochloric acid to one litre and standardize against sodium carbonate.

10.3 Procedure

10.3.1 Weigh accurately one gram of the sample into an iron crucible and fuse with about 5 g of sodium peroxide. Extract the melt in about 100 ml of water in a 400-ml beaker. Add about one to two gram of sodium peroxide to the water extract in the beaker, boil for about 5 minutes and filter. Wash the residue thoroughly with hot sodium hydroxide solution. Collect the filtrate and washings in the same beaker.

10.3.2 Neutralize the above filtrate with dilute hydrochloric acid providing an excess of 20 ml of concentrated hydrochloric acid. Add 10 ml of ferric chloride solution. Set the whole solution for evaporation, drying and baking. Extract the baked mass in about 20 ml of concentrated hydrochloric acid, dilute, filter, and wash free from acid by means of hot water. Boil and bring down the volume of the filtrate and the washings to about 100 ml. Transfer the solution to a 500-ml conical flask.

10.3.3 Neutralize the hot filtrate (80° to 100°C) with concentrated ammonium hydroxide until a slight persistent precipitate is formed. Re-dissovle the precipitate in a few drops of concentrated nitric acid providing finally about 2 ml of nitric acid in solution in excess. If the sample contains vanadium, add 2 ml of hydroxylamine hydrochloride solution. Add about 10 g of ammonium nitrate. Maintain the temperature of the solution between 40° to 50°C. Add about 40 ml of ammonium molybdate solution. Shake the flask vigorously for about 10 minutes and let stand for about 30 to 45 minutes.

10.3.4 Filter the precipitate through a thick pad of filter paper pulp prepared on a perforated disc in a funnel. Wash the beaker and the precipitate two to three times with dilute nitric acid and then with potassium nitrate wash solution till 10 ml of the filtrate collected in a test tube does not consume more than one drop of sodium hydroxide solution using a drop of phenolphthalein indicator solution (begin testing the filtrate from third wash). Transfer the pad of paper pulp with the precipitate back to the flask in which phosphorus was precipitated and wash down any precipitate adhering to the funnel with a jet of water, into the flask. Add 50 to 60 ml of water and add with stirring sodium hydroxide solution from a burette until all the yellow precipitate has dissolved and the solution is colourless. Add about 5 ml of sodium hydroxide solution in excess and stir. Add three to four drops of phenolphthalein indicator solution and titrate with standard hydrochloric acid until the pink colour is completely discharged.

10.3.5 Carry out a blank determination on the same quantity of sodium hydroxide solution which was added in the test, and titrating with standard hydrochloric acid, using three to four drops of phenolphthalein indicator solution.

10.4 Calculation

$$\text{Phosphorus Pentoxide, percent} = \frac{(A-B) \times C \times 0{\cdot}309}{D}$$

where

A = volume in ml of standard hydrochloric acid required for the blank test,

B = volume in ml of standard hydrochloric acid required for the sample,

C = normality of standard hydrochloric acid, and

D = weight in g of the sample taken.

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